#### GB829602

# Title: Improved method of producing alumina

### Abstract:

Aluminium hydroxide is calcined in the presence of a metallic halide and a boron compound to form an alumina which may be capable of being compacted to an increased degree before firing to form a refractory or a high temperature electrical insulator. The aluminium hydroxide may be produced by the Bayer process. The preferred halide is aluminium fluoride; chlorides, bromides and lodides may also be used, calcium and potassium halides being mentioned. The preferred boron compound is boric acid. When using aluminium fluoride and boric acid, preferred proportions are respectively up to 5% and 1-5% by weight of the aluminium hydroxide. Suitable calcining temperatures are 1400-1500 DEG C.

#### PATENT SPECIFICATION

NO DRAWINGS

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# COMPLETE SPECIFICATION

# Improved method of producing Alumina

We, General Motors Limited, a British Company, of 23, Buckingham Gate, London, S.W.I, do hereby declare the invention, for 5. W.1, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to an improved method of producing alumina from aluminium hydroxide, particularly from aluminium hydroxide which is produced by the Parent

hydroxide which is produced by the Bayer

process.

Calcined alumina is used as the main constituent in the manufacture of refractories. 15 electrical insulators such as those in spark plugs, and like articles. During manufacture of such articles the alumina is usually mixed with minor proportions of other materials. then moulded to shape, and finally fired, 20 when a certain amount of shrinkage occurs. One object of the present invention is to keep the shrinkage to a minimum and substantially constant for different batches of alumina mixtures: if the shrinkage is itself small, 25 then the possible relative shrinkage of different batches is very small and its effect becomes of secondary importance. The shrinkage depends partly on the degree to which the alumina mixture is com-

30 pacted during moulding: the greater the degree of compacting, the smaller the shrink-

age during firing.

Thus crystalline alumina which itself can be highly compacted is advantageous for 35 two reasons; firstly because shrinkage during firing is small, and secondly because possible variations in shrinkage between different batches of alumina are minimised.

A method according to the present inven-40 tion comprises calcining aluminium hydroxide in the presence of a metallic halide and a boron compound. By this method, the degree to which the crystalline alumina and hence the mixture to be moulded can be com-45 pacted can be increased.

The metallic halide may be a chloride,

bromide or iodide but is advantageously a fluoride.

If a metallic bromide or iodide is used, the degree to which the mixture can be com- 50 pacted is but slightly increased. Better results are obtainable with metallic chlorides but these are not as good as the results obtained with a metallic fluoride.

The metallic salt may, for example, be a 55 calcium or potassium salt, but is preferably an aluminium salt since such a salt, on decomposition during the calcination, not only does not leave any undesirable residue in the alumina but also increases the degree by 60 which the mixture can be compacted.

The boron compound is preferably boric

The quantities of the halide required to produce crystalline alumina which can be 65 compacted to the desired degree depends on the nature of the compound and the halide and on the temperature and time of calcina-

A certain molecular weight of fluorine ion 70 has a greater effect on the degree to which the alumina can be compacted than equivalent molecular weights of other halide ions.

The degree to which the alumina can be compacted is increased by the temperature of 75 calcination and duration of calcination. If, however, the duration of calcination exceeds a certain optimum time crystal growth occurs, and a higher firing temperature is required to obtain fired alumina of the de- 80 sired density than when the optimum calcination time is not exceeded.

If boric acid and aluminium fluoride are used, the former should be present in a proportion between 1% and 5% by weight and 85 the latter up to 5% by weight of the aluminium hydroxide, e.g. as produced by the Bayer process.

Crystalline alumina which can be highly compacted can be obtained by adding 2% by weight of powdered boric acid and 0.5% by weight of powdered aluminium fluoride

to aluminium hydroxide, and then calcining the mixture for three hours at a temperature of about 1,500° C. If the same mixture is calcined for three hours at 1,400°C, the degree to which alumina can be compacted

is appreciably reduced; and is markedly reduced when calcined for three hours at

Crystalline alumina which can also be 10 highly compacted can be obtained by adding 2% by weight of powdered boric acid and 1.0% by weight of powdered aluminium fluoride to aluminium hydroxide which has been produced e.g. by the Bayer process, and 15 then, as before, calcining the mixture for three hours at a temperature of 1,500° C. However, if the same mixture is calcined for three hours at 1,400° C. there is no appreciable change in the degree to which the alumina can be compacted. Thus a variation between 1,400° C. and 1,500° C. in the temperature at which different batches of this mixture are calcined does not affect the degree of shrinkage during firing after com-

Alumina produced according to this invention may advantageously be used for high temperature refractories and high temperature electrical insulators such as spark plugs.

WHAT WE CLAIM IS:

1. A method of producing alumina which comprises calcining aluminium hydroxide in the presence of a metallic halide and a boron

compound.

2. A method according to Claim 1 in 35 which the metallic halide is a fluoride.

3. A method according to Claim 1 or Claim 2 in which the metallic halide is an aluminium halide.

4. A method according to any of the pre- 40 ceding claims in which the boron compound

is boric acid.

5. A method of producing alumina which comprises calcining aluminium hydroxide which has been produced by the Bayer pro- 45 cess, in the presence of 1% to 5% of boric acid and up to 5% of aluminium fluoride by weight of the aluminium hydroxide.

6. A method of producing alumina which comprises calcining aluminium hydroxide 50 which has been produced by the Bayer process, in the presence of 2% boric acid and 1% of aluminium fluoride by weight of the

aluminium hydroxide.

7. A method according to Claim 6 in 55 which the aluminium hydroxide is calcined at a temperature of between 1,400° C. and 1,500° C. for three hours.

8. A high temperature refractory containing alumina produced by a method accord- 60

ing to any of the preceding claims.

9. A spark plug insulator containing alumina produced by a method according to any of Claims 1 to 7.

E. WILLIAMSON, Chartered Patent Agent.

## PROVISIONAL SPECIFICATION

# Improved method of producing Alumina

No. 29,616, A.D. 1957

We, GENERAL MOTORS LIMITED, a British Company, of 23, Buckingham Gate, London, S.W.1, do hereby declare this invention to be described in the following statement:—
This invention relates to an improved

70 method of producing alumina from raw

aluminium hydroxide by calcination.

Calcined alumina is used as the main constituent in the manufacture of refractories, electrical insulators such as those in spark 75 plugs, and like articles. During manufacture of such articles the alumina is usually mixed with minor proportions of other materials, then moulded to shape, and finally fired, when a certain amount of shrinkage occurs.

One object of the present invention is to keep the shrinkage to a minimum and substantially constant for different batches of alumina mixtures: if the shrinkage is itself small, then the possible relative shrinkage of different batches is very small and its effect becomes of secondary importance.

The shrinkage depends partly on the degree to which the alumina mixture is compacted during moulding: the greater the 90 degree of compacting, the smaller the shrink-

age during firing.

Thus crystalline alumina which itself can be highly compacted is advantageous for two reasons; firstly because shrinkage during firing is small, and secondly because possible 95 variations in shrinkage between different batches of alumina are minimised.

It has been discovered that such variations are likely to occur due to different times and temperatures of calcination.

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A method according to the present invention comprises reacting aluminium hydroxide during the calcination with a metallic fluoride and with a boron compound. By this method, the degree to which the crystal- 105 line alumina and hence the mixture to be moulded can be compacted can be increased.

The metallic fluoride may, for example, be calcium fluoride, but is preferably aluminium fluoride since this, on decomposition during 110 the calcination, does not leave any undesirable residue in the alumina.

The boron compound is preferably boric

acid. The quantities of the boron compound 115 and the fluoride required to produce crystalline alumina which can be compacted to the desired degree depends on the nature of the

compound and the fluoride and on the temperature and time of calcination. If boric acid and aluminium fluoride are used, the former should be present in a proportion 5 between 1% and 5% by weight and the latter up to 5% by weight of the aluminium hydroxide.

Crystalline alumina which can be highly compacted can be obtained by adding 2% by 10 weight of powdered boric acid and 0.5% by weight of powdered aluminium fluoride to powdered aluminium hydroxide, and then calcining the mixture for three hours at a temperature of about 1,500° C. If the same 15 mixture is calcined for three hours at 1,400° C, the degree to which alumina can be compacted is appreciably reduced; and is markedly reduced when calcined for three hours at 1,300° C.

O Crystalline alumina which can also be highly compacted can be obtained by adding 2% by weight of powdered boric acid and 1.0% by weight of powdered aluminium fluoride to the raw aluminium hydroxide, and then, as before calcining the mixture for three hours at a temperature of 1,500° C. However, if the same mixture is calcined for three hours at 1,400° C. there is no appreciable change in the degree to which the alumina can be compacted. Thus a variation between 1,400° C. and 1,500° C. in the temperature at which different batches of this mixture are calcined does not affect the degree of shrinkage during firing after compacting.

Alumina produced according to this invention may advantageously be used for high temperature refractories and high temperature electrical insulators such as spark plugs.

E. WILLIAMSON, Chartered Patent Agents.

# PROVISIONAL SPECIFICATION

No. 36,293, A.D. 1957

# Improved method of producing Alumina

We, GENERAL MOTORS LIMITED, a British Company, of 23, Buckingham Gate, London, S.W.1, do hereby declare this invention to be described in the following statement:—

This invention relates to an improved 45 method of producing alumina from raw aluminium hydroxide by calcination.

In the Provisional Specification accompanying our Application No. 29,616/57 there is described a method of producing 50 alumina from raw aluminium hydroxide by calcination which comprises reacting aluminium hydroxide during the calcination with a metallic fluoride and with a boron compound.

The present invention is based on the discovery that the other metallic halides, that is metallic chlorides, bromides and iodides, can be used instead of a metallic fluoride.

Therefore the method, according to the present invention, comprises reacting aluminium hydroxide during the calcination with a metallic chloride, bromide or iodide and with a boron compound.

These three metallic halides also increase 65 the degree to which the crystalline alumina and hence the mixture to be moulded can be compacted.

However if a metallic bromide or iodide is used, the degree to which the mixture can be compacted is but slightly increased. Better results are obtainable with metallic chlorides but these are not as good as the results obtained with a metallic fluoride.

The metallic salt may, for example, be a calcium or potassium salt, but is preferably an aluminium salt since such a salt, on decomposition during the calcination, not only does not leave any undesirable residue in the alumina but also increases the degree by which the mixture can be compacted.

The invention disclosed in this and the Provisional Specification accompanying Application No. 29,616/57 may be broadly described as a method of producing alumina from raw aluminium hydroxide by calcination which comprises reacting aluminium hydroxide during the calcination with a metallic halide and with a boron compound.

E. WILLIAMSON, Chartered Patent Agent,

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